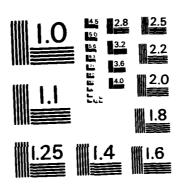
QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR MINUTEMAN. (U) DEGEN AIR LOGISTICS CENTER HILL AFB UT PROPELLANT ANALYSIS LR. J A THOMPSON OCT 84 MANPA-580(84) F/G 19/7 RD-R159 488 1/1 UNCLASSIFIED NL



MICROCOPY RESOLUTION TEST CHART
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HEADQUARTERS

OGDEN AIR LOGISTICS CENTER

UNITED STATES AIR FORCE

HILL AIR FORCE BASE, UTAH 84056

QUALIFICATION OF A NEW MAPO SOURCE AND ERL-510 CURING AGENT FOR MINUTEMAN STAGE I UF-2121 LINER

PROPELLANT ANALYSIS LABORATORY

MANPA REPORT

NR 500(84)

GCTOBER 1984



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QUALIFICATION OF A NEW MAPO

SOURCE AND ERL-510 CURING AGENT

FOR MINUTEMAN STAGE I UF-2121 LINER

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ABSTRACT

Thickol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thickol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first through the ninth

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time testing of the control and special specimens at Ogden ALC.

Statistical analysis of the test data showed that the physical properties of the new source compared closely to the old source of UF-2121 liner specimens.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.



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INTRODUCTION

A. PURPOSE:

Quality assurance testing of specimens prepared from the new source of MAPO to assure that liner material for First Stage Minuteman Motors will perform as predicted.

B. BACKGROUND:

tris [1-(2 methyl) aziridinyl] phosphine oxide (MAPO) is used as a curing agent in the Minuteman Stage One UF-2121 liner. MAPO was produced by Immont Chemical (Immont) and shipped to Arsynco Incorporated (Arsynco) for purification and marketing. Immont sold the production rights for MAPO to Arsynco and terminated the production of raw MAPO in 1972.

Since MAPO is a critical ingredient in UF-2121 liner formulation, the source change for the manufacture of MAPO was considered a serious change. Therefore, it was necessary for Thiokol to conduct qualification testing on liner material using MAPO manufactured by Arsynco before it could be considered acceptable for use in Minuteman Stage I UF-2121 liner.

ERLA-500 was the qualified epoxy curing agent used with MAPO in the UF2121 liner. Union Carbide terminated their process for ERLA-500. ERLA-510 used in similar liners (i.e. UF-2137) was substituted for ERLA-500 and qualified with MAPO from the new source.

The test conditions and test methods are shown in Table I.

*TWR-7857 Rev A Report, J. W. Rabern

Qualification testing was performed by Thiokol and reported in TWR-7857 Rev A. In addition, specimens were prepared by Thiokol From new vendor's material and from the old source material for a "follow on" test program. These specimens were then transferred to Ogden ALC for a continuing surveillance test program designed to cover a ten year span. This has been extended to 12 years as shown below in the 12 year contingency sample code. The material from the old source will be used as the control samples.

The 12 year sampling plan is shown below. Those specimens identified for the tenth year were tested at this test period. The types of specimens are Disc (steel/liner/steel), Cup (steel/liner/TP-H1011), and Peel (broad-cloth/liner/TP-H1011). For the disc specimen, the adhesion between the liner and steel is the critical factor. For the cup specimen, the adhesion between the propellant and the liner is critical. For the peel specimen, the propellant to liner peel strength when pulled at 180°F is critical.

TWELVE YEAR CONTINGENCY AGING SAMPLE CODING

Stora	ge						
Age	Temp	Disc (San		Cup (Sam		Peel (Sa	
<u>(yr)</u>	(°F)	Control*	Special**	Control*	Special**	Control*	Special**
1	75	1-6	181-186	61-66	241-246	121-126	301-306
2	75	7-12	187-192	67-72	247-252	127-132	307-312
3	75	13-18	193-198	73-78	253-258	133-138	313-318
4	75	19-24	199-204	79 – 84	259~264	139-144	319-324
5	75	25-30	205-210	85-90	265-270	145-150	325-330
6	75	31-36	211-216	91-96	271-276	151-156	331-336
7	75	37-42	217-222	97-102	277-282	157-162	337-342
9	75	43-48	223-228	103-108	283-288	163-168	343-348
10	75	49-54	229-234	109-114	289~294	169-174	349-354
12	75	55-60	235-240	115-120	295-300	175-300	355-360

^{*} Liner mix A73-11846 - Control or old MAPO source material

^{**} Liner mix A73-11810 - Experimental MAPO

TABLE I
Test Conditions and Methods

Group	Test	Condition	Config- uration	GO85 Spec Code	Spec Per Cond	Total Number of Spec	Test Method
Bond in Tension Disc	Tensile Adhesion OI#127-3	CHS 0.5 in/min, Chart 5.0 in/min, 500 lbs full scale load 77°F + 2°	Discs	TV	Control 6 Special 6	12	.
Bond in Tension Cup	Tensile Adhesion OI#127-3	CHS 0.5 in/min, Chart 5.0 in/min, 200 lbs full scale load 77°F + 2°	Cup	TC	Control 6 Special 6	12	A
180° Pee1 Specimens	Tensile Peel OI#127-3	CHS 10 in/min 77°F ± 2° Chart 5 in/ min 20 1bs full scale load	Peel	TE	Control 6 Special 6	12	В

TEST CONDITIONS

A. Testing of tensile adhesion specimens was performed using an Instron testing instrument. Properties measured were maximum stress to the nearest pound and failure mode.

Steel disc specimens require a stress of about 240 psi. The recommended initial full scale load is 500 pounds. This instrument setting should be changed to another setting if the first reading goes off scale on the high side. If 500 psi is exceeded, then change the reading to 1000 psi full scale.

Cup adhesion specimens are tested with a stress of 200 lbs per sq inch; the recommended full scale load is 500 lbs.

B. Testing of 180° peel samples was performed using an Instron testing instrument. The physical property of the material to be determined is the average peel strength to the nearest pound per inch.

NOTE: Thiokol's procedure for Testing and Laboratory Mixing of UF-2121 Liner. SLP 400, 28 April J1.

STATISTICAL ANALYSIS

UF-2121 liner material is being tested under a twelve year program to determine whether or not differences exist between liner materials manufactured from two separate sources of curing agent (MAPO). Test specimens were manufactured in two groups; control, using original source curing agent, and special, using new source curing agent. The test specimens for these two groups are three kinds: disc, cup, and peel. For each specimen type within each test group the sample test size is six. Laboratory testing for nine test periods or ten years has been accomplished. Test data for the years 1975 through 1984 are contained in Tables 2 through 10, and columns are summarized using means and standard deviations.

With nine test periods accomplished, regression plots (figures 1 thru

6) were made to determine whether slope and elevation differences existed
between control and special test data by using the analysis of covariance.

The regression model Y = a + bX, using individual data points, was used in
the regression analyses. The variance about the least squares trend line
is used to compute a tolerance interval such that at the 90% confidence level
90% of the sample distribution falls within this interval. This tolerance
interval is extrapolated 24 months past the age point pertaining to the oldest
specimens tested. The statistical significance of the slope of the trend line
is evaluated for each regression plot. If significant, it is an indication
that change over time is occurring.

In determining differences is data pertaining to the two MAPO sources, analysis of covariance was employed to compare control and special data from the regressions for each of the three types of test specimens. For analysis of covariance results, see table 11. Taken at the five percent significance

level, the only difference found was in the disc data in the variance of the data away from the regression line.

An in-house statistical investigation of the high - low, see - saw data pattern at every other month has been conducted. All test samples in this quality assurance program were prepared and labeled for testing at Thiokol, then transferred to Ogden ALC for testing. An error in labeling has been considered and tested.

The data was isolated and recombined with all the high months grouped together as comming from the same parent population, and all low months were also grouped together. New regression lines were put through each isolated data grouping. The isolated high new source regression trend lines were then compared against the high old source trend lines for combinability, or both isolated sets of data comming from the same parent population. All isolated regression trend lines were compared using the Analysis of Covariance testing.

The results showed that the residual variances about the isolated regression trend lines are considerably tighter. The trend line slopes are almost an overlay of each other. The trend line elevations however are out of the 95% confidence level. The isolated trend lines are not completely comparable, therefore a labeling error could not be varified.

TEST RESULTS

The 1984 test data and the means for the respective control and special data are shown in table 10. In addition, for a convenient comparison, the 1975 through 1983 test data are included in tables 2 through 9.

The statistical analysis results for the 1984 testing are shown in table 10 with the regressions shown in figures 1 through 6.

DISC:

A statistically significant difference is shown for variance of test data in the MAPO source (table 11).

The regression curves show a statistically significant gradual decrease for both the control and special sample data (figures 1 and 2).

For 1984, the mean of the control and special data is 15.22 and 15.35 kg/sq cm respectively.

The minimum specification requirement according to TWR-7857 Rev A, is 12.30 kg/sq cm minimum. As seen in table 10, MANPA's data is above this minimum.

The failure mode was 100% cohesive in the liner for both the control and special specimens.

CUP:

There is no significant difference in variance, slope or elevation when comparing control and special regression data (table 11).

The regression curves show a statistically significant gradual decrease in maximum stress as the specimens age (figures 3 and 4).

According to TWR-7857 Rev A Report, the minimum requirement is 4.92 kg/sq cm. The data means are 9.44 kg/sq cm for the control and 9.58 kg/sq cm for the special specimens.

For the control specimens, the failure mode was 100% in the propellant on all specimens. For the special specimens, three specimens broke 100% in the propellant and three specimens broke 95% in the propellant with 5% at the propellant to liner interface.

PEEL:

No significant difference is shown for the variance, slope or elevation when comparing control with special regression data (table 11).

The regression curves (figures 5 and 6) show a statistically significant increase in peel strength with respect to the age of the specimens.

Thiokol reported (TWR-7857 Rev A) 0.679 and 0.732 kg/L* cm respectively for the control and special mean data at age six months. This compares with 1984 data of 0.81 and 0.80 kg/l cm respectively for control and special mean data (table 10).

The mode of failure was 100% liner to propellant bond.

* Kilogram per linear centimeter.

CONCLUSIONS

Based on this analysis, the only statistically significant difference between the control and special specimens is the variance for disc specimens.

The disc and cup regressions show a gradual statistically significant decrease with the peel specimen data showing a statistically significant increase.

The strength of the specimens is well above the required minimum for disc and cup, and above that reported in Thiokol's testing for peel.

From the analysis of the data, the new source of raw material performs as well as the old source; and therefore is expected to perform satisfactorily.

RECOMMENDATIONS

It is recommended that the testing plan be continued to assure long range capability of the liner produced from the new source of material.

TABLE 2. TEST DATA SUMMARY FOR JULY 1975

DISC				UP	PEEL			
Control Special		Control Special		Control	Special			
Nr	Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm		
1	17.085	181 17.225	61	241 11.601	121 0.6786	301 0.6786		
2	17.507	182 17.858	62 11.812	242 11.601	122 0.6786	302 0.6965		
3	17.225	183 17.015	63 11.741	243 11.531	123 0.7143	303 0.6965		
4	17.929	184 16.944	64 12.163	244 11.671	124 0.7500	304 0.6965		
5	17.366	185 17.436	65 12.234	245 11.390	125 0.7679	305 0.7143		
6	17.296	186 19.054	66 11.882	246 11.390	126 0.7858	306 0.6965		
Ŧ	17.401	17.589	11.966	11.531	0.7292	0.6965		
S	0.2943	0.7899	0.2191	0.1176	0.0458	0.0113		

TABLE 3. TEST DATA SUMMARY FOR MAY 1976

DISC				UP	PEEL			
C	ontrol	Special	Control	Special	Control	Special		
Nr	Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cn	Nr Kg/L-cm		
-	15.116 16.311	187 14.483 188 14.905	67 68 13.710	247 13.288 248 13.710	127 0.9643 128 0.9286	307 1.0536 308 1.0358		
10 11	15.397 15.960 15.819 14.554	189 14.483 190 14.765 191 15.468 192 14.765	69 13.640 70 13.007 71 13.148 72 13.499	249 13.640 250 13.077 251 13.359 252 13.499	129 0.9286 130 1.0179 131 1.1072 132 1.0001	309 1.0179 310 1.0358 311 1.0536 312 1.0358		
y s	15.526 0.6356	14.812 0.3633	13.401 0.3088	13.429 0.2354	0.9911 0.0675	1.0388 0.0134		

TABLE 4. TEST DATA SUMMARY FOR APRIL 1977

	DISC			CUP				PEEL			
Co	ontrol	Special	Control Speci			cial				pecial	
	Kg/cm ²	Nr Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cn ²		Kg/L-cm	Nr	Kg/L-cm	
13	17.155	193 17.436	73	9.281	253	9.140		0.7322		0.7858	
14	16.522	194 16.874	74	9.281	254	9.070	134	0.7143	314	0.7858	
15	16.874	195 16.944	75	8.999	255	8.999	135	0.6786	315	0.7858	
16	17.015	196 17.366	76	9.281	256	9.140	136	0.7500	316	0.7143	
17	16.874	197 17.015	77	9.492	257	8.367	137	0.7500	317	0.7500	
-	16.874	198 17.015	78	9.281	258	8.789	138	0.6429	318	0.7143	
Ÿ	16.886	17.108		9.269		8.918		0.7113		0.7560	
S	0.2107	0.2337		0.1570		0.2994		0.0429		0.0352	

NOTE: Kg/L-cm = Kilograms per linear centimeter. Average peel is given for each peel parameter.

TABLE 5. TEST DATA SUMMARY FOR JUNE 197	TABLE	5.	TEST	DATA	SUMMARY	FOR	JUNE	1978
---	-------	----	------	------	---------	-----	------	------

DISC			C	UP	PEEL			
Control Special		Control	Special	Control	Special			
Nr	Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm		
19	14.906	199 14.554	79 10.898	259 10.406	139 0.9109	319 1.0180		
20	14.624	200 14.695	80 10.968	260 10.476	140 0.9823	320 1.0359		
21	14.695	201 14.343	81 10.617	261 10.616	141 0.9466	321 1.0180		
22	14.906	202 14.343	82 10.125	262 10.125	142 1.0002	322 1.0359		
23	14.343	203 14.624	83 10.406	263 10.687	143 1.0716	324 1.0537		
24	15.187	204 14.414	84 10.476	264 10.125	144 0.9466	326 0.9287		
Ŧ	14.777	14.496	10.582	10.406	0.9764	1.0150		
S	0.2898	0.1503	0.3166	0.2391	0.0561	0.0444		

TABLE 6. TEST DATA SUMMARY FOR JUNE 1979

		*************	THE DATA DOISELT TON O				<u> </u>			
	DI	SC		C	UΡ			PEI	EL_	
C	ontrol	Special	Control Spec		cial	Co	ontrol	S	Special	
Nr	Kg/cm ²	Nr Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/L-cm	Nr	Kg/L-cm
25	17.015	205 17.436	85	9.632	265	9.562	145	0.8572	325	0.7679
26	16.874	206 16.874	86	9.703	266	9.140	146	0.8393	326	0.7679
27	16.874	207 16.593	87	9.773	267	9.562	147	0.8572	327	0.9643
28	16.944	208 16.522	88	9.632	268	9.281	148	0.8572	328	0.7143
29	16.804	209 16.382	89	9.632	269	9.421		0.7679	329	0.7143
30	16.171	210 17.366	90	9.492	270	9.421	150	0.8036	330	0.7500
<u>y</u>	16.780	16.862		9.644		9.398		0.8304		0.7798
S	0.3070	0.4477		0.0934		0.1644		0.0370		0.0936

TABLE 7. TEST DATA SUMMARY FOR SEPTEMBER 1980

	DISC	CI	JP	PEI	EL	
Control Special		Control	Special	Control	Special	
Nr Kg/cr	n ² Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/cm ²	Nr Kg/L-cm	Nr Kg/L-cm	
31 15.11	16 211 14.764	91 9.281	271 11.109	151 0.9465	331 1.1965	
32 14.90	05 212 14.413	92 10.054	272 10.265	152 1.0179	332 1.1786	
33 14.62	24 213 14.202	93 10.616	273 10.898	153 1.0715	333 1.1072	
34 14.90	05 214 14.272	94	274 10.687	154 1.1072	334 1.0536	
35 15.6	78 215 13.870	95 9.894	275 9.491	155 1.0179	335 1.0358	
36 15.11	16 216 14.272	96 10.054	276	156 1.0715	336 1.0358	
¥ 15.0	57 14.284	9.998	10.490	1.0388	1.0846	
S 0.35	0.3188	0.4750	0.6397	0.0569	0.0613	

TABLE	8.	TEST	DATA	SUMMARY	FOR	JUNE	1984

DISC					CUP				PE	EL	
C	ontrol	Sı	pecial	Co	Control		Special		Control	Sı	pecial
Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²
37	14.905	217	16.874	97	9.421	277	8.999	157		337	0.8036
38	15.819	218	16.944	98	9.210	278	9.070	158	0.7679	338	0.9018
39	15.116	219	15.187	99	9.281	279	9.351	159	0.7858	339	0.8126
40	15.749	220	16.101	100	9.351	280	8.437	160	0.8393	340	0.8304
41	15.890	221	15.679	101	9.632	281	9.562	161	0.8483	341	0.8572
42	15.468	222	15,608	102	9.351	282	9.843	162	0.8215	342	0.8929
Y	15.491		16.066		9.374		9.210		0.8126		0.8929
S	0.4044		0.7151		0.1452		0.4911		0.0346		0.0413

TABLE 9. TEST DATA SUMMARY FOR SEPTEMBER 1983

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/cm ²										
43	14.056	223	14.786	103	10.792	283	10.188	163	1.0358	343	1.2144
44	14.995	224	13.758	104	10.870	284	10.771	164	0.9822	344	1.1608
45	14.309	225	14.190	105	10.715	285	10.940	165	1.0179	345	1.2144
46	14.309	226	12.058	106	10.448	286	11.109	166	1.1251	346	1.2501
47	14.905	227	13.668	107	10.462	287	10.842	167	1.17.86	347	1.1786
48	14.220	228	14.086	108	11.067	288	10.377	168	1.2144	348	1.0715
	14.466		13.758		10.726		10.704		1.0923		1.1816
	0.3874		0.9219		0.2402		0.3511		0.0941		0.0623

TABLE 10. TEST DATA SUMMARY FOR JUNE 1984

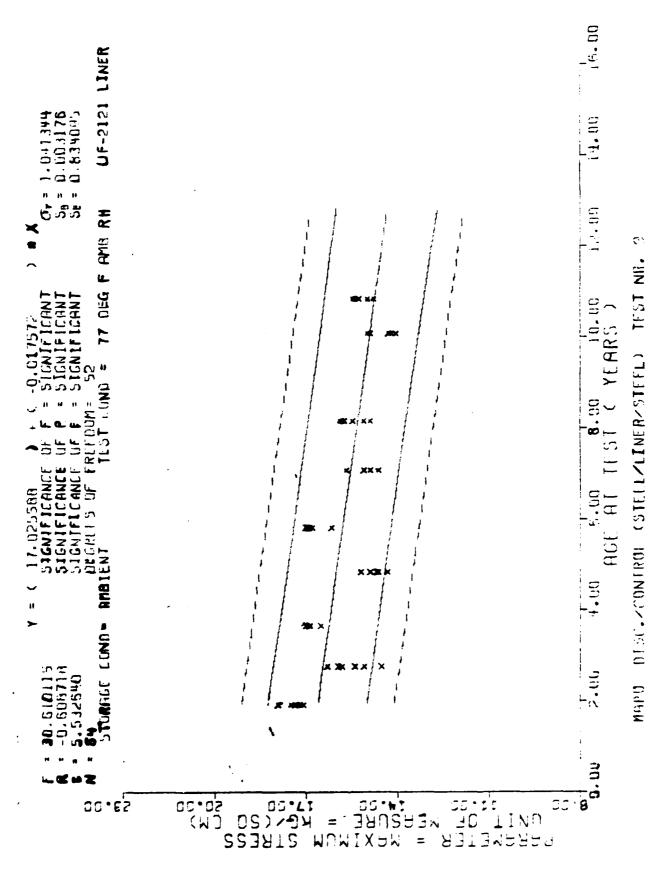
DISC. DIST.												
DISC			CUP					PEEL				
	Control	S	pecial	C	ontrol	S	pecial		Control		Special	
Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	Nr	Kg/cm ²	
49	14.780	229	14.980	109	9.140	289	9.280	169	*	349	0.770	
50	15.260	230	14.980	110	10.120	290	10.410	170	*	350	0.800	
51	15.400	231	15.470	111	9.630	291	9.630	171	0.820	351	0.790	
52	15.400	232	16.310	112	9.140	292	9.490	172	0.820	352	0.840	
53	15.470	233	15.120	113	9.420	293	9.420	173	0.800	353	0.790	
54	14.980	234	15.260	114	9.140	294	9.280	174	0.800	354	0.790	
	15.215		15.353		9.437		9.585		0.810		0.797	
	0.2757		0.5041		0.3921		0.4254		0.016		0.023	

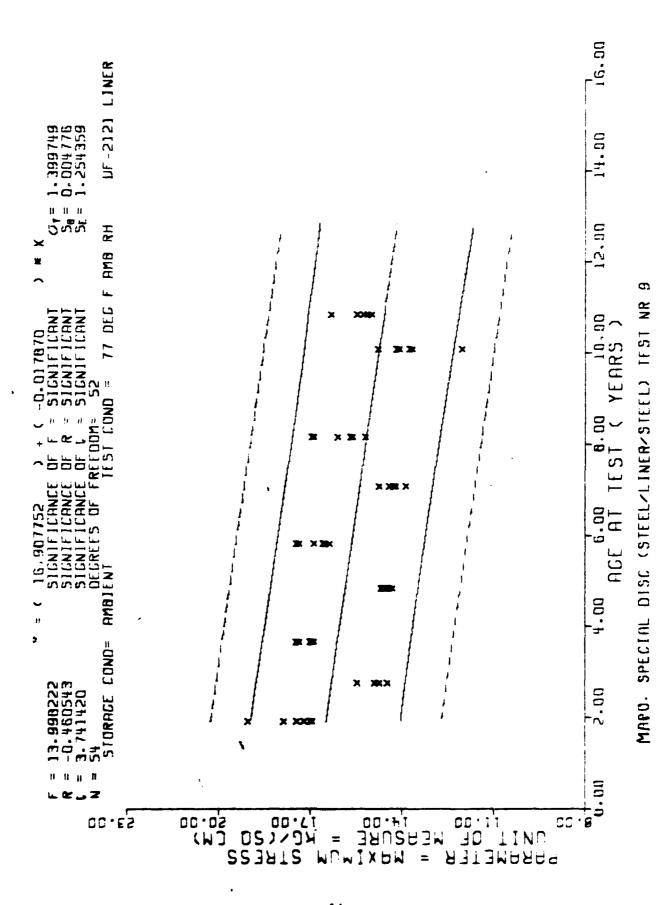
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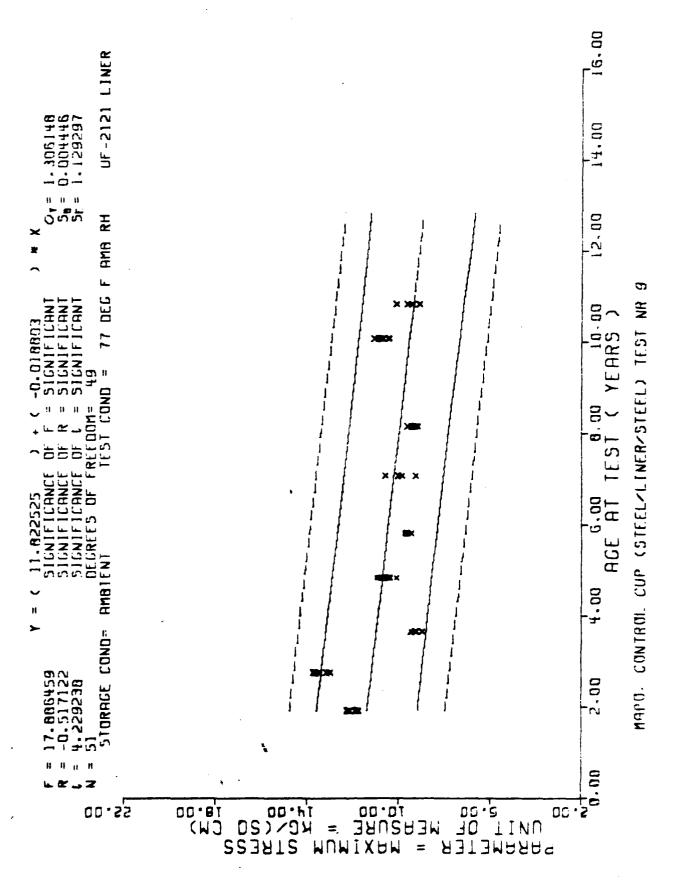
TABLE 11. ANALYSIS OF COVARIANCE RESULTS COMPARING CONTROL AND SPECIAL REGRESSION DATA

Type of Data Compared	Parameter Compared	F-value	Degrees of Freedom	Covariance Results
	Variance	2.39	52,52	Significant
DISC	Slope	0.01	1,104	Not Applicable
	Elevation	0.49	1,105	Not Applicable
	Variance	1.25	51,49	Not Significant
CUP	Slope	0.10	1,100	Not Significant
	Elevation	0.03	1,101	Not Significant
	Variance	1.50	52,49	Not Significant
PEEL	Slope	0.00	1,101	Not Significant
	Elevation	0.18	1,102	Not Significant

NOTE: The regressions are compared using the Analysis of Covariance at the 5% significance level. If comparison between residual variances is significant, further testing is not applicable.







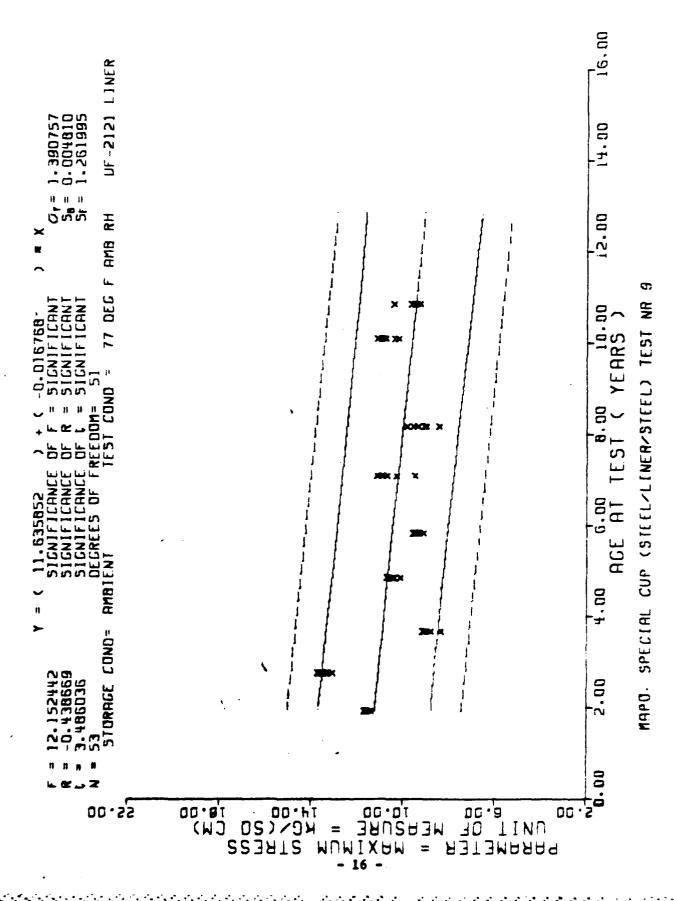
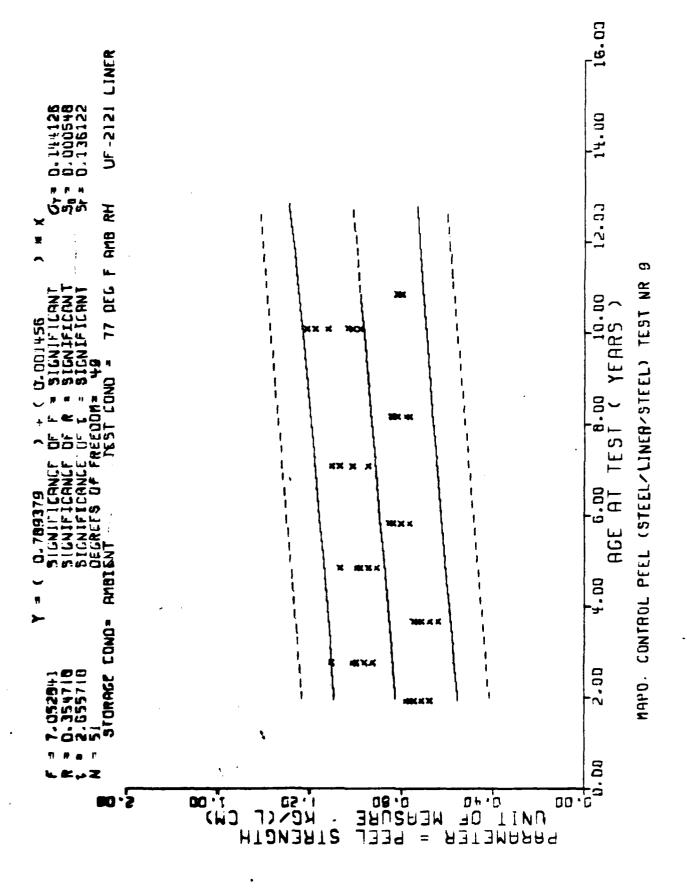
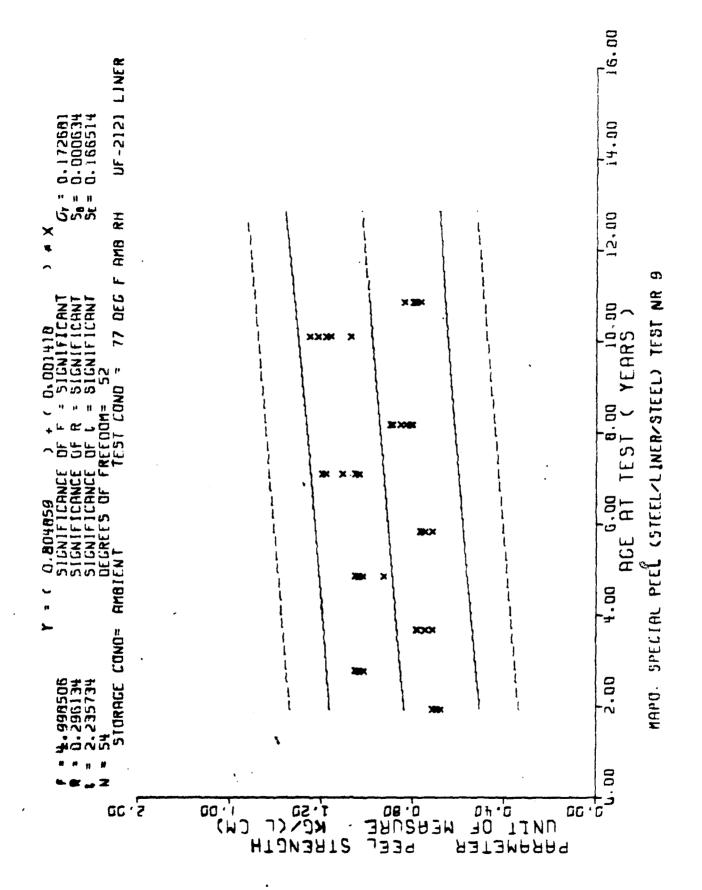


Figure 4





REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 2. GOVT ACCESSION NO.	RECIPIENT'S CATALOG NUMBER
MANPA REPORT NR 500(84)	29480
4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED
Qualification of a new MAPO source and ERL-510	m == D == 14= C==d A====1
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UF-2121 liner. Thiokol changed MAPO vendors and, th	nerefore, qualification of the
new source MAPO was required. Thiokol prepared specimens from the new source	and also enecimens from the
original source which are to be used as the control	
veillance testing program. The specimens were trans	
testing and reporting of the data obtained.	

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